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PROCESS FEASIBILITY STUDY IN SUPPORT OF SILICON MATERIAL TASK I

QUARTERLY TECHNICAL PROGRESS REPORT (VII)

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Technical Content Statement

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ABSTRACT

During this reporting period, major activities were continued on process system properties, chemical engineering and economic analyses for application to alternate processes under consideration for solar cell grade silicon.

In Task 1, primary efforts were devoted to properties of silicon source materials. For silane, liquid viscosity data were correlated as a function of temperature to cover the entire liquid range. Estimates for gas and liquid thermal conductivity are reported. Unfortunately, there are no experimental data available for thermal conductivity of silane.

Correlation results for silane are also presented for heat and free energy of formation of the gas as a function of temperature. Both American and Russian data sources were used in the correlation. Correlation and data values are in good agreement with average deviations being less than 0.30 kcal/g-mol.

For Task 2, major efforts were devoted to preliminary process design for the conventional polysilicon process based on the rod reactor (hairpin) technology. Engineering design calculations for the preliminary process flowsheet, material balance and energy balance are essentially 100% complete. Major process equipment design and production labor requirements are 80% complete. Initial results indicate that the key items are M.G. silicon and HCl consumption: high electrical requirements for the rod reactors; and the large number of major equipment items (62). The criteria for design were selected to be commensurate with the design basis for the alternate processes to produce solar cell grade silicon.

Chemical engineering analysis activities in Task 2 also focused on preliminary process design for a silane (SiH_4) plant. A revised flowsheet received from Union Carbide has received a preliminary review, and process design has been reinitiated.

In Task 3, preliminary cost analysis is in progress for the conventional polysilicon process used in the United States and Europe for the production of semiconductor grade polysilicon. Plant investment and product cost estimates will be determined upon completion of review of major process equipment and production labor requirements.

TABLE OF CONTENTS

I. PROCESS SYSTEM PROPERTIES ANALYSES (TASK 1)

Major activities for process system properties of silicon source materials were devoted to property data required in the performance of the chemical engineering analyses of the alternate process under consideration for solar cell grade silicon production.

For silane, liquid viscosity data are available (A30) in the temperature range between the melting point and boiling point. The data were extended to cover the entire liquid range with the following correlation (A63) for viscosity of the saturated liquid as a function of temperature:

$$\log \mu_L = A + B/T + CT + DT^2 \qquad \text{(I-1)}$$

In Eq. (I-1), μ_L = viscosity of saturated liquid, centipoise; A,B,C,D = correlation constants characteristic for the chemical compound and T = temperature, $^{\circ}K$.

Correlation values and data were in good agreement with average absolute deviation of 1.4%. Results for liquid viscosity versus temperature are given in Figure I-1.

Unfortunately, there are no experimental data available for gas thermal conductivity of silane. In the absence of data, gas thermal conductivity for silane was estimated by the modified Eucken correlation for polyatomic gases:

$$k_G = \mu_G \left[1.32\ C_p + \frac{0.891}{M}\right] \qquad \text{(I-2)}$$

where k_G = gas thermal conductivity at low pressure, cal/(sec)(sm)($^{\circ}K$); μ_G = gas viscosity, poise; C_p = gas heat capacity, cal/(g) ($^{\circ}k$); and M = molecular weight, g/g-mol.

The Eucken correlation results agrees well with values of Svehla (A40); deviations are less than 1%. Figure I-2 presents results for gas thermal conductivity.

Liquid thermal conductivity results for silane are given in Figure I-3. The liquid thermal conductivity for silane was estimated with the modified Stiel and Thodos (A29) relation:

$$k_L = \frac{f(\rho_r)}{\gamma Z_c^5} + k_G \qquad \text{(I-3)}$$

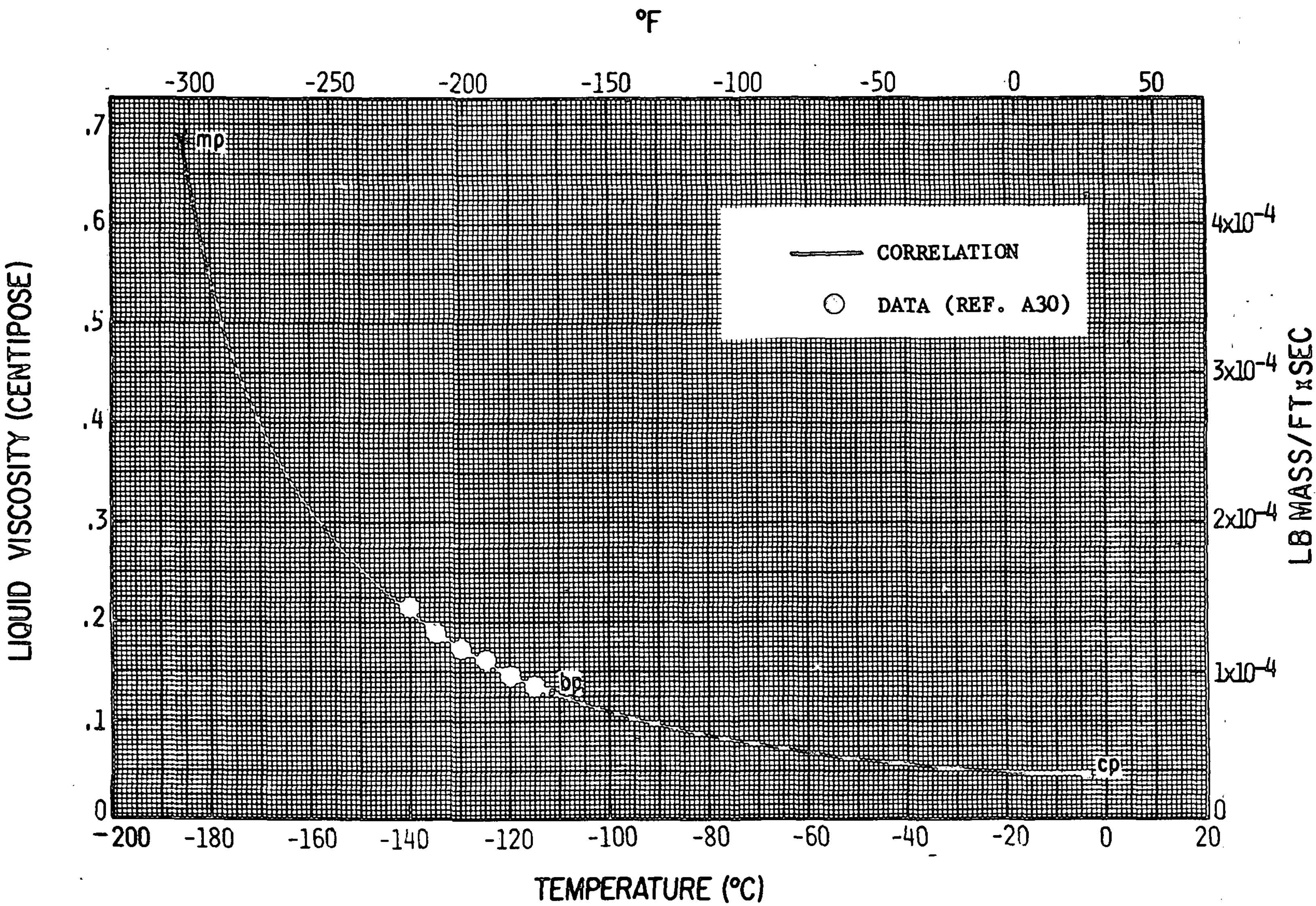

Figure I-1 Liquid Viscosity Vs. Temperature for Silane

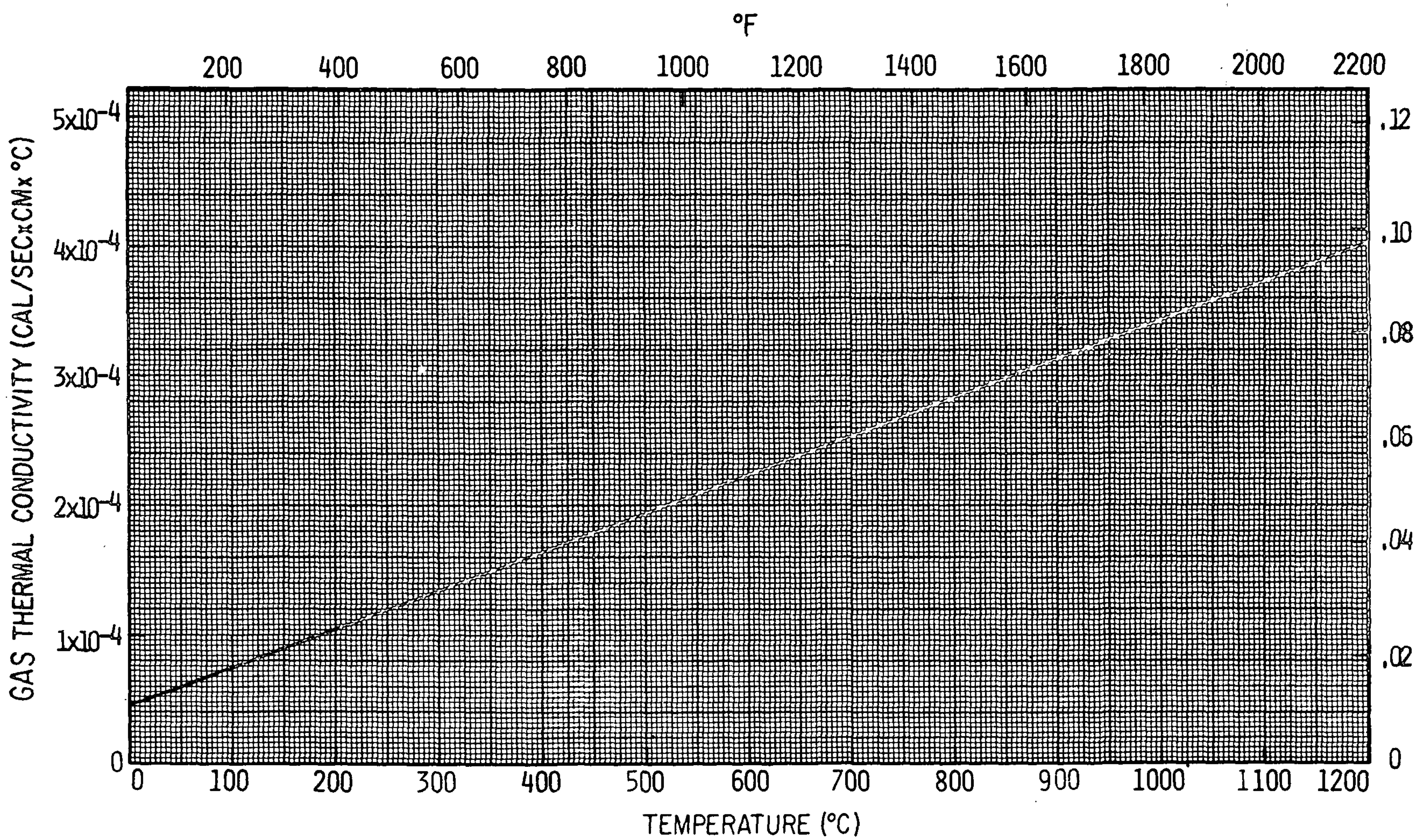

Figure I-2 Gas Thermal Conductivity Vs. Temperature for Silane

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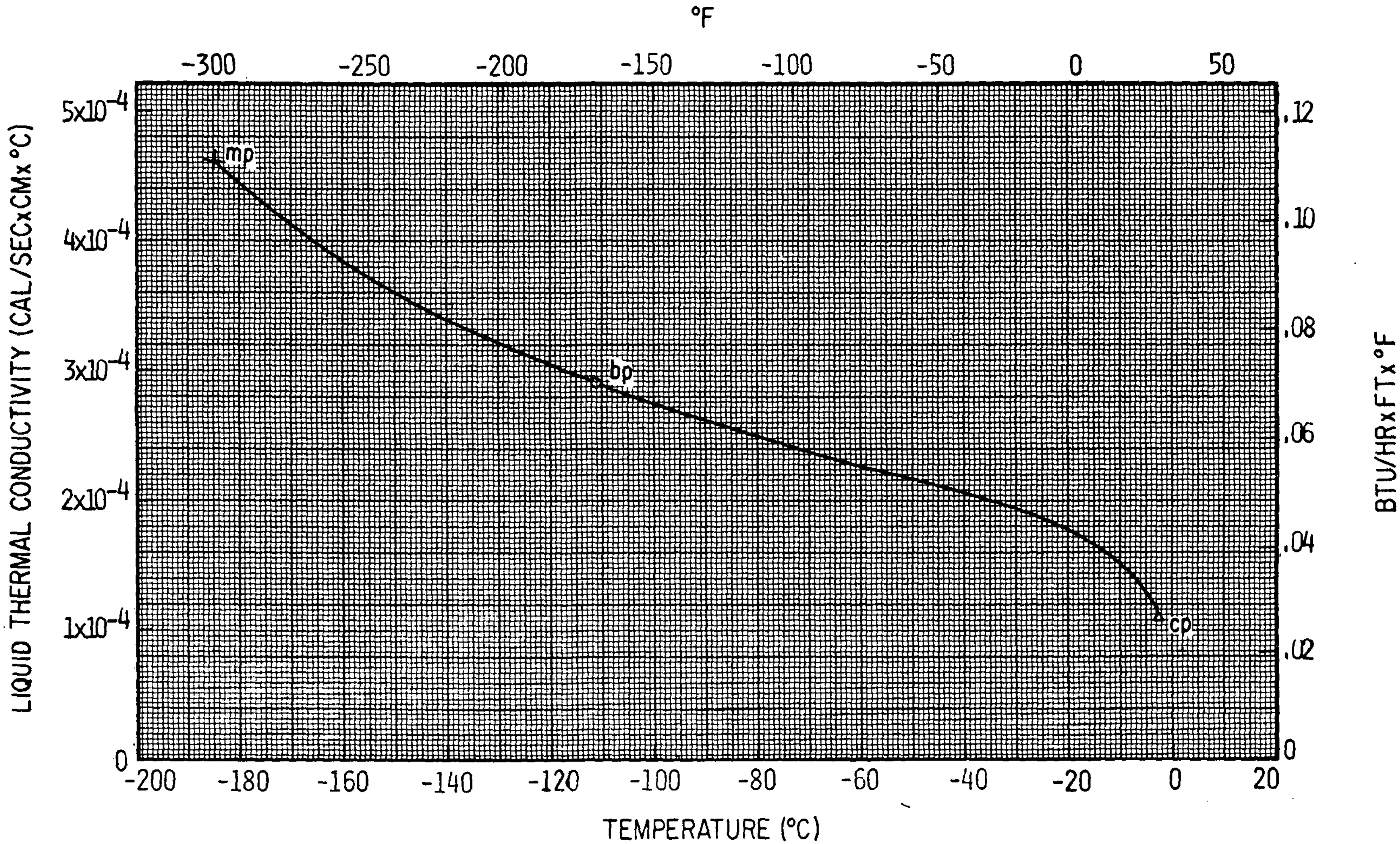


Figure I-3 Liquid Thermal Conductivity Vs. Temperature for Silane

where k_L = liquid thermal conductivity, cal/(sec)(cm)(^oK); k_G = gas thermal conductivity at low pressure (1 atm), cal/(sec)(cm)(^oK); $\gamma = T_c^{1/6} M^{1/2}/P_c^{2/3}$; ρr = reduced density, $\rho/\rho c$; and Z_c = critical compresibility factor.

The correlation was tested with experimental data for methane with average deviations of less than 17%. The deviations for silane are probably in the same range. The presented results are intended to represent correct order-of-magnitude values.

Data for heat of formation of silane are available from American and Russian sources (A12, A39). These data were correlated for heat of formation of the ideal gas by a series expansion in temperature:

$$\Delta H_f = A + BT + CT^2 \qquad \text{(I-4)}$$

where ΔH_f = heat of formation of ideal gas at low pressure, kcal/g-mol; A,B,C = correlation constants characteristic for the chemical compound; and T = temperature, ^oK.

A least squares regression analysis of the available data was used to determine the constants A,B, and C. A generalized least-squares regression computer program for minimizing deviation was used to process the numerous data points.

Correlation and data values are compared in Figure I-4 for silane. The agreement is quite good. In most cases, the average absolute deviations of correlation and data are less than 0.30 kcal/g-mol.

Data for free energy of formation are also available from American and Russian investigations (A12, A39) for silane.

Correlation constants for free energy of formation of the ideal gas were based on a linear relationship in temperature.

$$\Delta G_f = A + BT \qquad \text{(I-5)}$$

where ΔG_f = free energy of formation of ideal gas at low pressure, kcal/g-mol; A, B = correlation constants characteristic of the chemical compound; and T = temperature, ^oK.

The correlation constants, A and B, were ascertained from a least-squares regression analysis of the available data. The regression analysis was done with a generalized least-squares computer program for minimizing deviation.

Correlation and data values for free energy of formation compare favorably, as illustrated in Figure I-5 for silane. Average absolute deviations between correlation and data values are less than 0.30 kcal/g-mol in most cases.

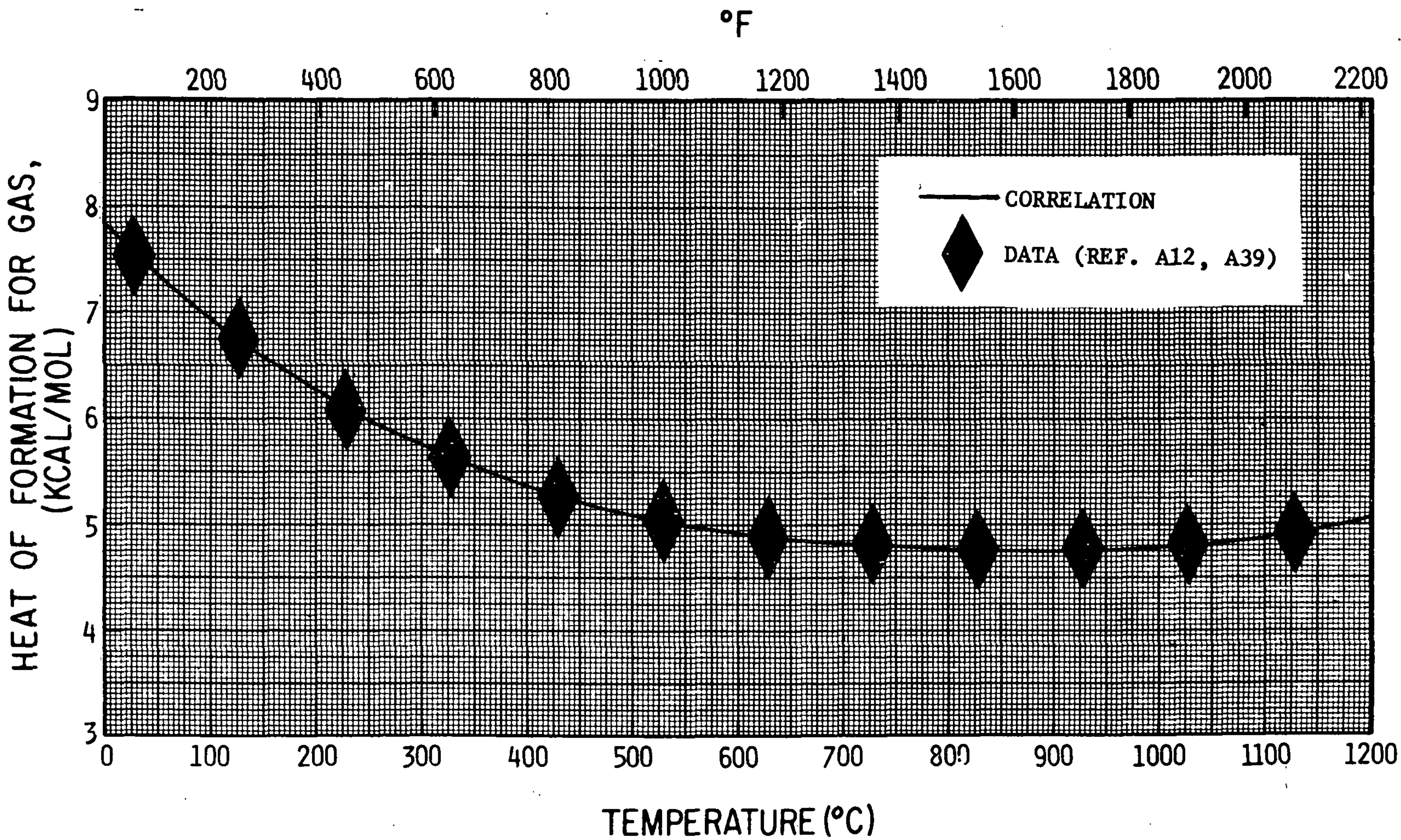


Figure I-4 Heat of Formation Vs. Temperature for Silane

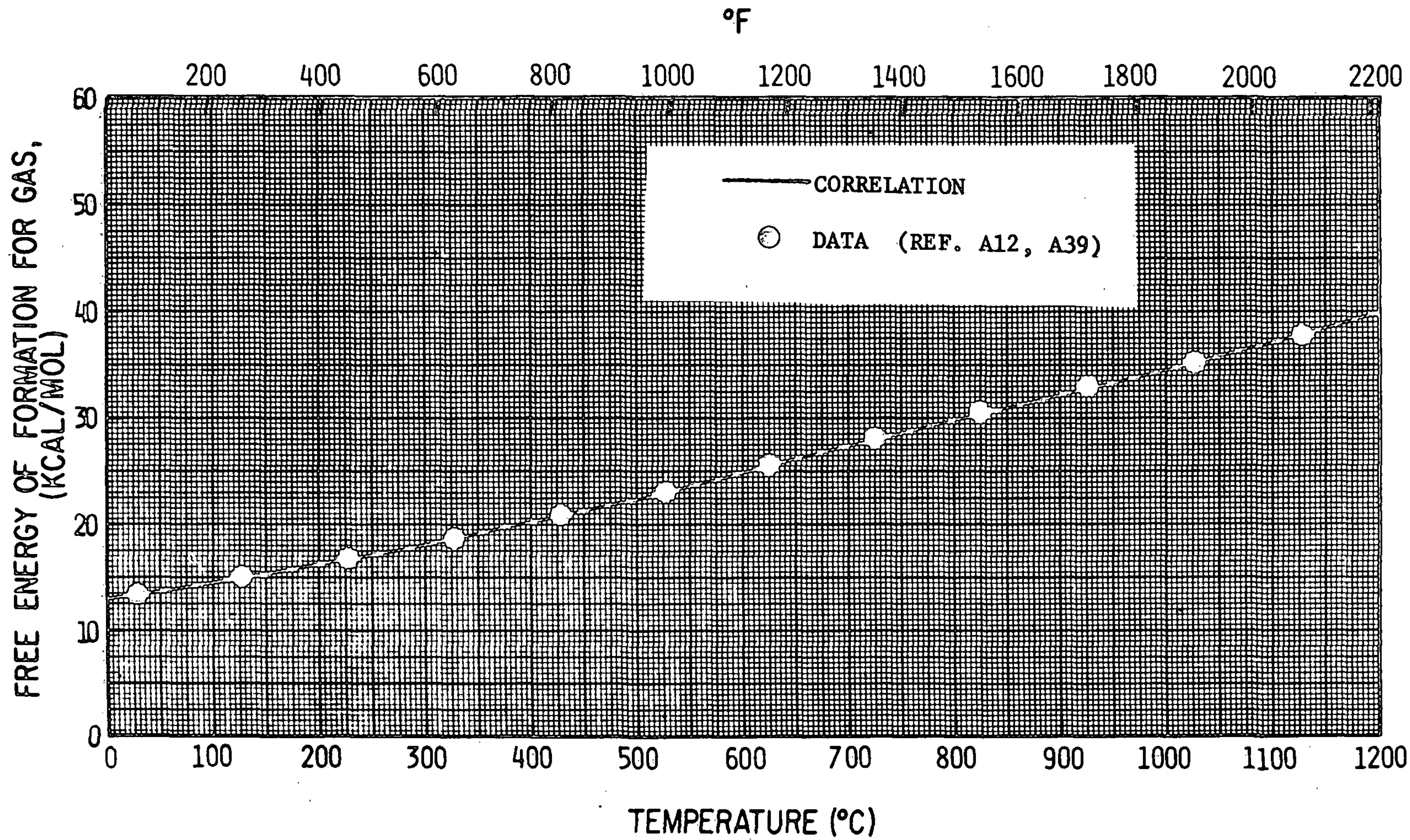


Figure I-5 Free Energy of Formation Vs. Temperature for Silane

II. CHEMICAL ENGINEERING ANALYSES (TASK 2)

A. Silane Process (Union Carbide)

Major effors during this reporting period, were devoted to the preliminary process design for the silane process (Union Carbide). The status, including progress since the last reporting period for the process design is given below for key guideline items:

	Current
Process Flow Diagram	75%
Material Balance	50%
Energy Balance	0%
Property Data	40%
Equipment Design	10%

The status, including activities accomplished, in progress, and planned are shown in Table IIA-1 for the preliminary process design.

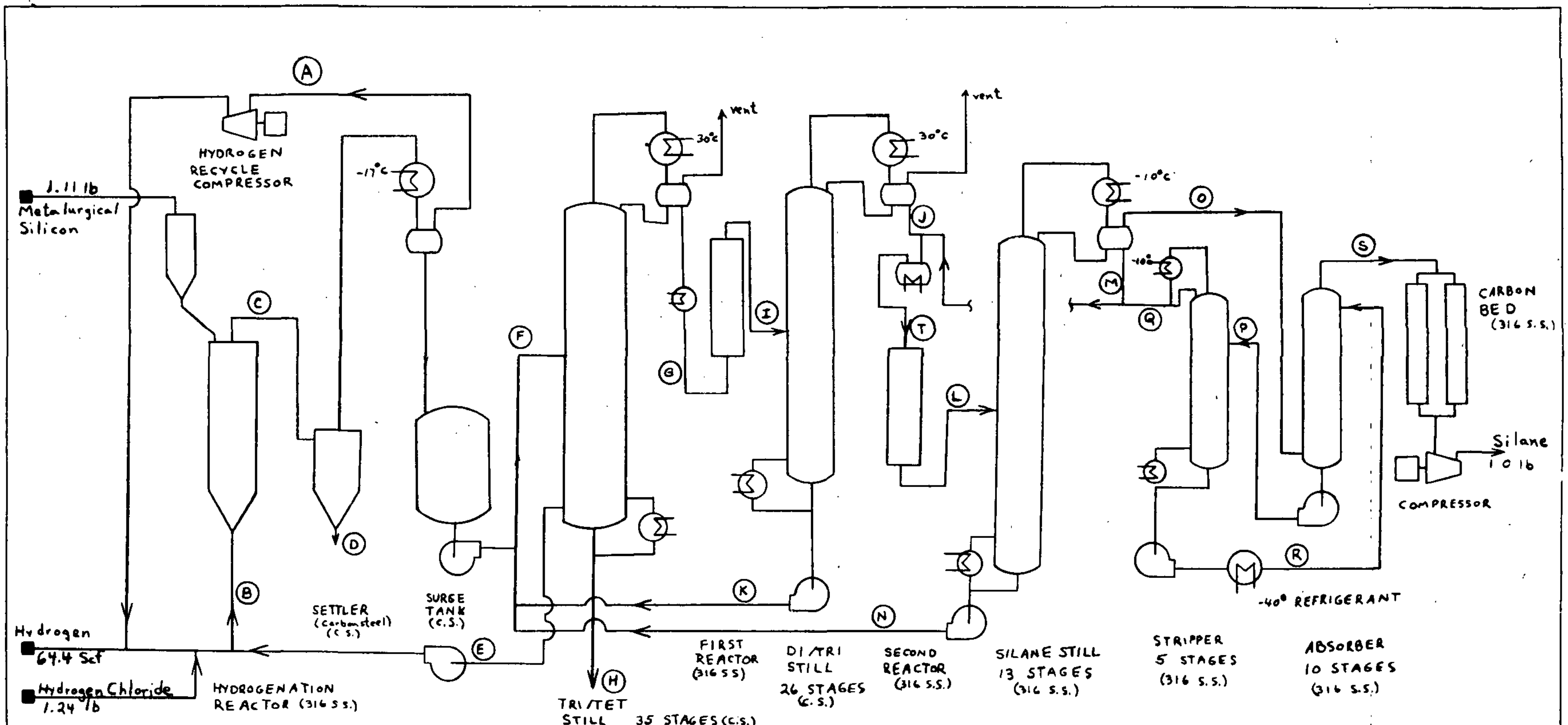
A revised flowsheet has been received by Mr. W. C. Breneman of Union Carbide. A preliminary review of the revised flowsheet is in progress. The revised flowsheet is shown in Figure IIA-1.

TABLE IIA-1 CHEMICAL ENGINEERING ANALYSES: PRELIMINARY PROCESS DESIGN ACTIVITIES FOR SILANE PROCESS (UNION CARBIDE)

Prel. Process Design Activity	Status
1. Specify Base Case Conditions	●
1. Plant Size	●
2. Product Specifics	●
3. Additional Conditions	●
2. Define Reaction Chemistry	◐
1. Reactants, Products	●
2. Equilibrium	◐
3. Process Flow Diagram	◐
1. Flow Sequence, Unit Operations	●
2. Process Conditions (T, P, etc.)	◐
3. Environmental	◐
4. Company Interaction (Technology Exchange)	◐
4. Material Balance Calculations	◐
1. Raw Materials	●
2. Products	●
3. By-Products	◐
5. Energy Balance Calculations	0
1. Heating	0
2. Cooling	0
3. Additional	0
6. Property Data	◐
1. Physical	◐
2. Thermodynamic	◐
3. Additional	◐

Prel. Process Design Activity	Status
7. Equipment Design Calculations	0
1. Storage Vessels	0
2. Unit Operations Equipment	0
3. Process Data (P, T, rate, etc.)	0
4. Additional	0
8. List of Major Process Equipment	0
1. Size	0
2. Type	0
3. Materials of Construction	0
8a. Major Technical Factors	0
(Potential Problem Areas)	0
1. Materials Compatibility	0
2. Process Conditions Limitations	0
3. Additional	0
9. Production Labor Requirements	0
1. Process Technology	0
2. Production Volume	0
10. Forward for Economic Analysis	0

0 Plan
◐ In Progress
● Complete



Ref. Point	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P	Q	R	S	T	U	V	
Temp., °C	-17	550	550	110	124		80	124	80	25	85	80	-5	85	-5	-5	-5	-40	-40	80			
Press., psig	50	50	45	45	50	50	50	50	50	50	50	45	45	50	45	35	35	45	45	45			
Flow, lb/hr.	6.6	139	140	.16	13.1	268	136	1.3	136	10.2	125	20.7	9.3	9.0	2.3	2.85	1.3	1.6	1.03	20.7			
H_2, mole %	94.7	49.4	45.9	-	-	-	-	-	-								-	-	-				
$SiCl_4$, mole %	3.6	49.2	43.3	80	99.0	42.8	-	99.5	11.5		12.8					35.4	-	97.5	.2				
$HSiCl_3$, mole %	1.7	.3	10.8	20	1.0	57.2	99.0	.5	78	1	87.2	29	4.4	91.2		-	-		-	2.5			
H_2SiCl_2, mole %	-	-	-		-	-	1.0		10	99		35	67.8	8.3	15.1	27.8	44.3		-	80.2			
H_3SiCl, mole %	-	-	-		-	-			0.5			16	18.4		16.8	31	47.6	2.5	.7	12.2			
SiH_4, mole %	-	-	-		-	-			-			20	9.4		67.6	5.1	8.1		99.1	5.1			
HCl, mole %		1.1	-		-	-																	

REF. DWG.

CHEMICALS AND PLASTICS
UNION CARBIDE CORPORATION
SISTERSVILLE, WEST VIRGINIA

Silicon to Silane Process Flowsheet

DR.	CH.	AP.	SCALE	DATE	
WCB				2-10-76	C-4334-2

Figure IIA-1 Process Flow Sheet for Silane Process (Revised, Provided by Union Carbide)

B. Conventional Polysilicon Process

All resources and a majority of manpower were committed to the preliminary process design for the conventional polysilicon process. In an effort to report initial results, routine review procedures have been circumvented. Therefore, these findings are subject to further review and modification. The status for the preliminary process design, including status since the last reporting period, is summarized below for major items:

	Prior	Current
Process Flow Diagram	90%	100%
Material Balance	90%	100%
Energy Balance	80%	100%
Property Data	20%	80%
Equipment Design	30%	80%
Production Labor	0%	80%

The detailed status for all components that make up the preliminary process design is given in Table IIB-1.0. The preliminary process flow-sheet is shown in Figure IIB-1.0.

The results are summarized in Table IIB-1.1 to IIB-1.6. The guide for these tables is given below:

Base Case Conditions	Table IIB-1.1
Reaction Chemistry	Table IIB-1.2
Raw Materials Requirements........	Table IIB-1.3
Utility Requirements..............	Table IIB-1.4
List of Major Process Equipment...	Table IIB-1.5
Production Labor Requirements.....	Table IIB-1.6

The base case conditions (Table IIB-1.1) were selected so that the designs and economic analyses prepared for alternate processes to produce solar cell grade silicon might be compared to the conventional polysilicon process. The preliminary design was prepared for an integrated plant with trichlorosilane (TCS) production, TCS purification, and semiconductor grade silicon production. In all cases, proven commercial technology was utilized; (1) fluidized bed utilizing metalurgical grade (M.G.) silicon and anhydrous HCl to produce TCS, (2) distillation for the purification of TCS (and by-product silicon tetrachloride, if desired), and (3) semiconductor grade poly-silicon via the Sieman's type rod (hairpin) reactor. The open literature (refs. 31, 32, and 20) were utilized to obtain exit gas compositions from both reaction systems, and the TCS conversion to silicon (growth rate) was obtained from reference 20. Technical interchange was maintained with Dr. Leon Crossman of Dow Corning Corporation. Storage considerations and operating ratio were selected commensurate with similar parameters for the alternate processes being reviewed.

TABLE IIB-1.0 CHEMICAL ENGINEERING ANALYSES:
PRELIMINARY PROCESS DESIGN ACTIVITIES FOR

Prel. Process Design Activity	Status
1. Specify Base Case Conditions	●
1. Plant Size	●
2. Product Specifics	●
3. Additional Conditions	●
2. Define Reaction Chemistry	●
1. Reactants, Products	●
2. Equilibrium	●
3. Process Flow Diagram	●
1. Flow Sequence, Unit Operations	●
2. Process Conditions (T, P, etc.)	●
3. Environmental	●
4. Company Interaction (Technology Exchange)	●
4. Material Balance Calculations	●
1. Raw Materials	●
2. Products	●
3. By-Products	●
5. Energy Balance Calculations	●
1. Heating	●
2. Cooling	●
3. Additional	●
6. Property Data	●
1. Physical	●
2. Thermodynamic	●
3. Additional	●

Prel. Process Design Activity	Status
7. Equipment Design Calculations	◒
1. Storage Vessels	◒
2. Unit Operations Equipment	◒
3. Process Data (P, T, rate, etc.)	◒
4. Additional	◒
8. List of Major Process Equipment	◒
1. Size	◒
2. Type	◒
3. Materials of Construction	◒
8a. Major Technical Factors (Potential Problem Areas)	◒
1. Materials Compatibility	◒
2. Process Conditions Limitations	◒
3. Additional	◒
9. Production Labor Requirements	◒
1. Process Technology	◒
2. Production Volume	◒
10. Forward for Economic Analysis	◒

O Plan
◒ In Progress
● Complete

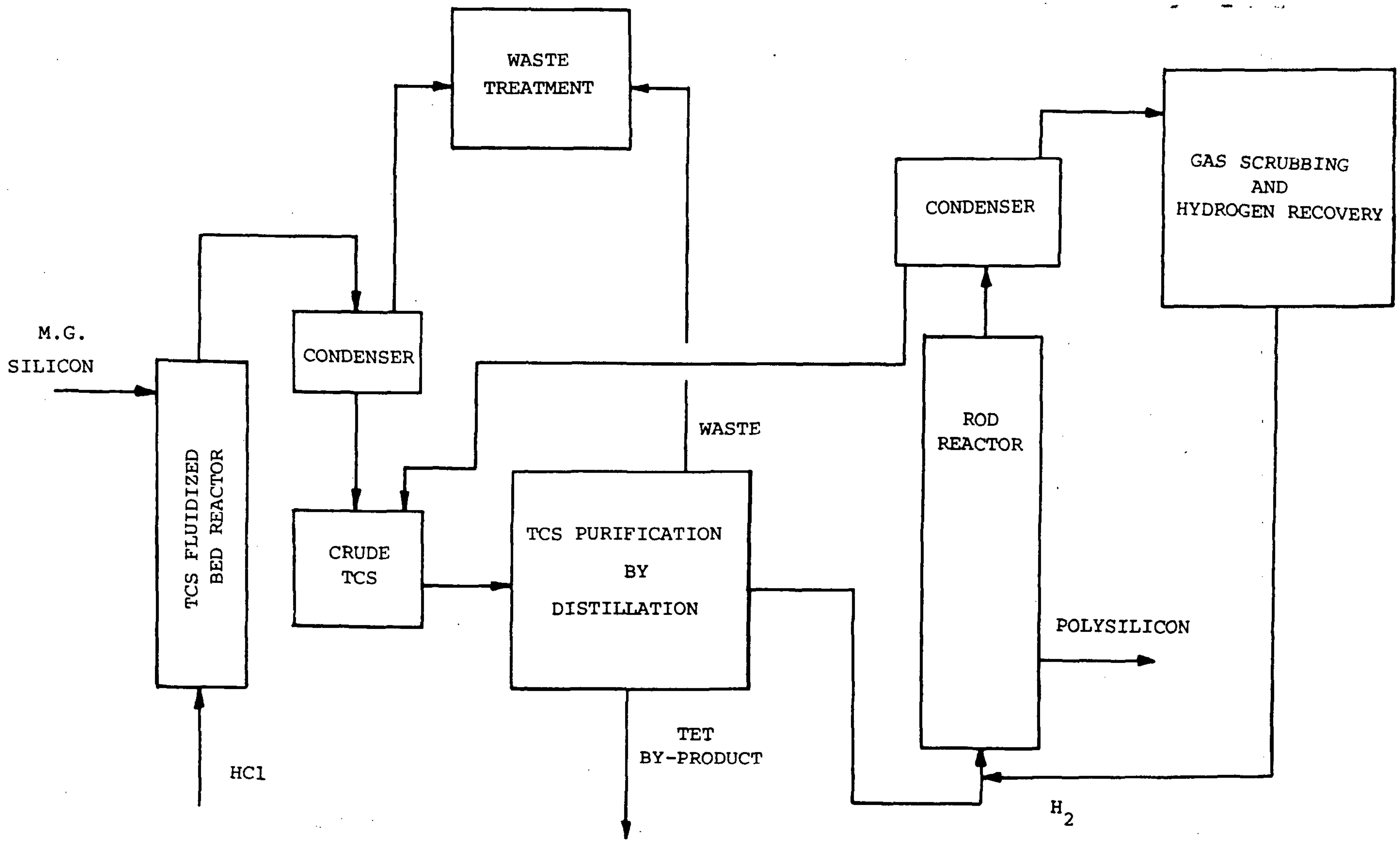


Figure III-1.0 Preliminary Process Flowsheet for Conventional Polysilicon Process

TABLE IIB-1.1

BASE CASE CONDITIONS FOR CONVENTIONAL POLYSILICON PROCESS

1. Plant Size
   - 1000 metric tons per year
   - Semiconductor grade silicon

2. Production of TCS
   - Fluidized Bed, 600°K, low pressure (65 PSIA)
   - Metallurgical grade silicon plus HCl gas
   - Chlorosilane content in condensed reator gas by moles (ref. 32)

     91.5% TCS ($SiCl_3H$)
     5.2% TET ($SiCl_4$)
     1.4% DCS ($SiCl_2H_2$)
     1.9% Heavies

   - Slight excess HCl in reator gas (1%)
   - Hydrogen burned

3. TCS Purification (ref. 31)
   - Distillation
   - 5% lights to waste (5% of TCS & TET)
   - Separate TCS and TET
   - 5% heavies from TCS & TET to waste
   - TET for by-product sales
   - TCS to rod reactor

4. Silicon Production
   - Rod reactor at 1050°C, 20 PSIA
   - Hydrogen to reduce TCS
   - Entering gas analysis

     10% TCS
     90% H_2

   - 8.17 moles TCS in/mole of S; production in an operating reactor
   - Exit gas analysis (ref. 20)

     4.339% TET
     4.457% TCS
     .089% DCS
     2.197% HCl
     88.92% H_2

5. Waste Treatment
   - Light and heavy cuts from distillation to waste treatment
   - Vapors from TCS reactor condenser to scrubber
   - Vapor from rod reactor to scrubber
   - All waste streams neutralized with NaOH

TABLE IIB-1.1 (Continued)

6. Recycles
   - H_2 from rod reactor dried and returned, 5% losses
   - Chlorosilanes from rod reactor condensed off gas recycled to purification (distillation)

7. Operating Ratio
   - Approximately 90% utilization
   - Approximately 7880 hour/year production

8. Storage Considerations
   - Feed materials (two week supply)
   - Product (two week supply)
   - Process (several days)

TABLE IIB-1.2

REACTION CHEMISTRY FOR CONVENTIONAL POLYSILICON PROCESS

1. TCS Reactor

$$Si + 3\,HCl \rightarrow SiHCl_3 + H_2$$
$$Si + 4\,HCl \rightarrow SiCl_4 + 2H_2$$
$$Si + 2HCl \rightarrow SiH_2Cl_2$$

2. Rod Reactor

$$SiHCl_3 + H_2 \rightarrow Si + 3HCl$$
$$SiHCl_3 + HCl \rightarrow SiCl_4 + H_2$$
$$SiHCl_3 + H_2 \rightarrow SiH_2Cl_2 + HCl$$

3. Waste Treatment

$$SiHCl_3 + 2H_2O \rightarrow SiO_2 + 3HCl + H_2$$
$$SiCl_4 + 2H_2O \rightarrow SiO_2 + 4HCl$$
$$SiH_2Cl_2 + 2H_2O \rightarrow SiO_2 + 2HCl + 2H_2$$
$$HCl + NaOH \rightarrow NaCl + H_2O$$

TABLE IIB-1.3

RAW MATERIAL REQUIREMENTS FOR CONVENTIONAL POLYSILICON PROCESS

	Raw Material	Requirement lb/Kg of Silicon
1.	M. G. Silicon	6.72 Kg/Kg
2.	Anhydrous HCl	57.96
3.	Hydrogen	.828
4.	Caustic (50% NaOH)	53.29
5.	$SiCl_4$ (By Product)	46.12

TABLE IIB-1.4

UTILITY REQUIREMENTS FOR
CONVENTIONAL POLYSILICON PROCESS

UTILITY/FUNCTION		REQUIREMENTS/Kg OF SILICON PRODUCT
1. Electricity		384.6 Kw-Hr
1. All pump motors (16 motors)	(.339)	
2. 2 compressor motors	(9.243)	
3. Polysilicon Rod Reactor	(375) (ref. 33)	
2. Steam (250 PSIA)		152 Pounds
1. HCl Vaporizer	(7.07)	
2. Caustic Storage Tank	(1.82)	
3. #1 Scrubber Vapor Heater	(.276)	
4. #1 Distillation Column Calandria	(38.75)	
5. #2 Distillation Column Calandria	(47.73)	
6. #3 Distillation Column Calandria	(25.24)	
7. TCS Vaporizer	(10.79)	
8. #2 Scrubber Vapor Heater	(3.4)	
9. Liquid Recycle Heater	(5.52)	
10. #4 Distillation Column Calandria	(11.3)	
11. Rod Reactor	(-1287 generated)	
3. Cooling Water		98.5 Gallons
1. TCS Reactor Off Gas Cooler	(13.91)	
2. Rod Reactor Off Gas Cooler	(334)	
3. #4 Distillation Column Condenser	(37.24)	
4. Polysilicon Rod Reactor Cooling End Plates	(473)	
5. TCS Reactor Off Gas Compressor	(11.12)	
6. Rod Reactor Off Gas Compressor	(115.2)	
4. Process Water		320.9 Gallons
1. #2 Gas Scrubber	(31.36)	
2. #1 Gas Scrubber	(134.82)	
3. To Make Steam In Cooling Rod Reactor Side Walls	(154.7)	
5. Refrigerant (-40°F)		42.1 M BTU
1. TCS Reactor Off Gas Condenser	(12.57)	
2. Rod Reactor Off Gas Condenser	(29.52)	
6. Refrigerant (34°F)		92.3 M BTU
1. #1 Distillation Column Condenser	(34)	
2. #2 Distillation Column Condenser	(37.4)	
3. #3 Distillation Column Condenser	(20.85)	
7. High Temperature Heat Exchange Fluid		582 Pounds
1. TCS Fluidized Bed Reactor	(581)	
2. Nitrogen Heater	(0.61)	
8. Nitrogen		349.1 SCF
1. Molecular Sieves	(328.5)	
2. Polysilicon Rod Reactor Purge	(20.64)	

TABLE IIB-1.5

LIST OF MAJOR PROCESS
EQUIPMENT FOR CONVENTIONAL POLYSILICON PROCESS

		Type	Function	Duty	Size	Materials of Construction
1.	(T1)	M.G. Silicon Storage Hopper	Raw Material Storage	2 Weeks Storage	6.5×10^4 gallons	CS
2.	(T2)	Liquid HCl Storage Tank	Raw Material Storage	2 Weeks Storage	2.5×10^5 gallons 250 PSIA	Nickel Steel
3.	(T3)	Crude TCS Hold Tanks (3)	Feed for Purification	1 Week Storage	2.77×10^5 gallons (each)	CS
4.	(T4)	Waste Hold Tank	Feed For Waste Treatment	1 Week Storage	3.025×10^4 gallons	CS
5.	(T5)	TCS Reactor Off Gas Flash Tank	Phase Separation		1 ft. in diameter by 4 ft. tall, 300 PSIA	SS
6.	(T6)	Hydrogen Storage Tank	Make-up For Losses	8 Hours Backup for Pipeline Failure	7.24×10^4 gallons Spherical 250 PSIA	CS
7.	(T7)	Polysilicon Storage Space	Final Product Storage	2 Weeks Storage	1300 ft.^{3} of space	CS
8.	(T8)	TET Storage Tanks (2)	Final By-product Storage	2 Weeks Storage	1.62×10^5 Gallons (each)	CS
9.	(T9)	TET Feed Tanks (2)	Feed for Distillation Column #4	1 Week Storage	8.83×10^4 Gallons (each)	CS
10.	(T10)	TCS Feed Tanks (3)	Feed for Distillation Column #3	1 Day Storage	2.47×10^4 Gallons (each)	CS
11.	(T11)	TCS Storage Tanks (3)	Purified TCS Hold-Up Feed to Rod Reactor	1 Week Storage	1.64×10^5 Gallons (each)	CS
12.	(T12)	TET/TCS Feed Tanks (3)	Feed for Distillation Column #2	1 Day Storage	3.75×10^4 Gallons (each)	CS

TABLE IIB-1.5 (continued)

13.	(T13)	Caustic Storage Tank	Raw Material Storage	2 Week Storage 1.91×10^5 BTU/HR	1.82×10^5 Gallons	SS
14.	(T14)	#1 Distillation Condenser Flash Tank	Phase Separation		1 Ft. in Diameter by 4 Feet Tall	CS
15.	(T15)	Rod Reactor Off Gas Flash Tank	Phase Separation		1 Ft. in Diameter by 4 Feet Tall 300 PSIA	SS
16.	(H1)	HCl Vaporizer	Vaporize Feed To TCS Reactor	7.5×10^5 BTU/Hr	38.29 $Ft.^2$ 250 PSIA Shell	SS/SS
17.	(H2)	TCS Reactor Off Gas Cooler	Cool Reaction Gas	4.4×10^5 BTU/Hr	224 $Ft.^2$ 65 PSIA Tubes	CS/SS
18.	(H3)	TCS Reactor Off Gas Condenser	Condense Reaction Gas	1.6×10^6 BTU/Hr	1423 $Ft.^2$ 300 PSIA Tubes	SS/SS
19.	(H4)	#1 Scrubber Vapor Heater	Heat Vapor Wastes to 40°F for Scrubbing	3×10^4 BTU/Hr	15.7 $Ft.^2$ 250 PSIA Shell	CS/SS
20.	(H5)	#1 Distillation Column Condenser	Condense Overheads for Relux	4.31×10^6 BTU/Hr	1540 $Ft.^2$	CS/SS
21.	(H6)	#1 Distillation Column Calandria	Reboiler for Column #1	4×10^6 BTU/Hr	311. $Ft.^2$ 250 PSIA Shell	CS/SS
22.	(H7)	#2 Distillation Column Condenser	Condense Overheads For Reflux	4.7×10^6 BTU/Hr	1555 $Ft.^2$	CS/CS
23.	(H8)	#2 Distillation Column Calandria	Reboiler for Column #2	5×10^6 BTU/Hr	402.4 $Ft.^2$ 250 PSIA Shell	CS/SS
24.	(H9)	#3 Distillation Column Condenser	Condense Overheads for Reflux	2.64×10^6 BTU/Hr	867 $Ft.^2$	CS/CS

TABLE IIB-1.5 (continued)

25.	(H10)	#3 Distillation Column Calandria	Reboiler for Column #3	2.64×10^{6} BTU/Hr	173 Ft.^{2} 250 PSIA Shell	CS/SS
26.	(H11)	TCS Vaporizer	Vaporize Feed To Rod Reactor	1.13×10^{6} BTU/Hr	73 Ft.^{2} 250 PSIA Shell	CS/CS
27.	(H12)	Rod Reactor Off Gas Cooler	Cool Reaction Gas	1.06×10^{7} BTU/Hr	2519 Ft.^{2} 20 PSIA	CS/SS
28.	(H13)	Rod Reactor Off Gas Condenser	Condense Reaction Gas	3.74×10^{6} BTU/Hr	3341 Ft.^{2} 300 PSIA Tubes	SS/SS
29.	(H14)	#2 Scrubber Vapor Heater	Heat Vapor Wastes to 40^{o}F for Scrubbing	3.56×10^{5} BTU/Hr	180 Ft.^{2} 250 PSIA Shell	CS/SS
30.	(H15)	Liquid Recycle Heater	Heat Cold Recycle Liquid (Crude TCS) to 80^{o}F for Storage	5.79×10^{5} BTU/Hr	30.6 Ft.^{2} 250 PSIA Shell	SS/SS
31.	(H16)	#4 Distillation Column Condenser	Condenser Overheads for Reflux	1.18×10^{6} BTU/Hr	513 Ft.^{2}	CS/CS
32.	(H17)	#4 Distillation Column Calandria	Reboiler for Column #4	1.18×10^{6} BTU/Hr	95 Ft.^{2} 250 PSIA Shell	CS/SS
33.	(H18)	Nitrogen Heater	Heat Regenerator Gas for Molecular Sieves	2.46×10^{4} BTU/Hr	44.8 Ft.^{2}	CS/CS
34.	(P1)	TCS Reactor Off Gas Compressor	Compress Reaction Gas For Condensation	3.52×10^{5} BTU/Hr	138.2 Horsepower	CS
35.	(P2)	Caustic Supply Pump	Supply Caustic for Waste Neutralization and Gas Scrubbers		9 gpm 100 Ft. of Head	SS
36.	(P3)	#1 Distillation Column Overheads Pump	Supply Reflux and Remove Waste to Waste Hold Tank		62.2 gpm 100 Ft. of Head	CS*

TABLE IIB-1.5 (continued)

37.	(P4)	#1 Distillation Column Calandria Pump	Forced Convection Pump		93 gpm 150 Ft. of Head	CS*
38.	(P5)	TET/TCS Feed Pump	Feed #2 Distillation Column		26.1 gpm 100 Ft. of Head	CS*
39.	(P6)	#2 Distillation Column Overheads Pump	Supply Relux, Pump Overhead to TCS Feed Tank		70 gpm 100 Ft. of Head	CS*
40.	(P7)	TCS Feed Pump	Feed #3 Distillation Column		21 gpm 100 Ft. of Head	CS*
41.	(P8)	#2 Distillation Column Calandria Pump	Forced Convection Pump		104 gpm 150 Ft. of Head	CS*
42.	(P9)	#3 Distillation Column Overhead Pump	Supply Reflux, Pump Overheads to TCS Storage Tank		39 gpm 100 Ft. of Head	CS*
43.	(P10)	Rod Reactor TCS Feed Pump	Feed TCS to Rod Reactor		15 gpm 100 Ft. of Head	CS*
44.	(P11)	#3 Distillation Column Calandria Pump	Forced Convection Pump		39 gpm 150 Ft. of Head	CS*
45.	(P12)	Rod Reactor Off Gas Compressor	Compress Reaction Gas for Condensation	3.65×10^6 BTU/Hr	1434 Horsepower	CS
46.	(P13)	#4 Distillation Column Overheads Pump	Supply Reflux Pump TET by product to TET Storage Tank		21.59 gpm 100 Ft. of Head	CS*
47.	(P14)	#4 Distillation Column Calandria Pump	Forced Convection Pump		22.4 gpm 100 Ft. of Head	CS*

NOTES

*Includes incremental higher cost for special purity requirements.

TABLE IIB-1.5 (continued)

48.	(P15)	TET Feed Pump	Feed #4 Distillation Column		9.2 gpm 100 Ft. of Head	CS*
49.	(P16)	Waste Treatment Pump	Pump from Waste Hold To Waste Treatment		2.8 gpm 50 Ft. of Head	CS
50.	(P17)	Crude TCS Feed Pump	Feed Purification Area		28 gpm 100 Ft. of Head	CS*
51.	(P18)	Process Water Feed Pump	Feed Process Water to Scrubber and Waste Treatment		350 gpm 100 Ft. of Head	CS
52.	(C1)	#1 Gas Scrubber	Scrub Gas Wastes from TCS Reactor Off Gas		43 Ft. Tall D = 3½ Ft.	SS
53.	(C2)	#2 Gas Scrubber	Scrub Gas Wastes from H16, H3, H5		40 Ft. Tall D = 2¼ Ft.	SS
54.	(C3)	#1 Distillation Column	Separate Light Impurities to Waste		29 Trays 24 inches apart 3 3/4 Ft. in Diameter	CS
55.	(C4)	#2 Distillation Column	Separate TET and TCS		29 Trays 24 inches apart 4¼ Ft. in Diameter	CS
56.	(C5)	#3 Distillation Column	Separate Heavies TCS to Waste		15 Trays 20 inches apart 3 Ft. in diameter	CS
57.	(C6)	#4 Distillation Column	Separate Heavies TET to Waste		15 Trays 20 inches apart 2¼ Feet in Diameter	CS
58.	(R1)	TCS Fluidized Bed Reactor	Production of TCS For Rod Reactor	4.552×10^{6} BTU/Hr (Cooling)	D = 2.61 Ft. L = 28.8 Ft. 64, 1" O D Cooling Tubes 9.4' Long	SS

TABLE IIB-1.5 (continued)

59. (R2)	Polysilicon Rod Reactors (285)	Production of Polysilicon		Hairpin Reactor (2 hair-pins, 3 Ft. long, 6 Inch Dia.)	Quartz
60. (A1)	Molecular Sieves (2)	Dry Out Rod Reactor Off Gas For Hydrogen Recycle		D = 3.5 Ft. L = 14.4 Ft.	CS
61. (A2)	Fines Separator	Remove Solids From Fluidized Bed Reactor Off Gas		12" Cyclone Separator	SS
62. (A3)	Hydrogen Flare	Dispose of Hydrogen Produced in TCS Fluidized Bed Reactor	8.94×10^6 BTU/Hr	30 Feet High Stack 6" diameter	CS

TABLE IIB-1.6

PRODUCTION LABOR REQUIREMENTS FOR
CONVENTIONAL POLYSILICON PROCESS

	Unit Operation	Type	Skilled Labor Man Hrs/Day	Skilled Labor Per Kg Si	Semiskilled Labor Per Day	Semiskilled Labor Per Kg Si
1.	TCS Production	A	80	.0292		
2.	Vaporization	B	60	.0219		
3.	Vapor Compression	B	60	.0219		
4.	Vapor Condensation	B	60	.0219		
5.	TCS/TET Separation	C	40	.0146		
6.	TCS Purification	C	35	.0128		
7.	TET Purification	C	30	.011		
8.	Waste Treatment	B	80	.0292		
9.	Gas Scrubbing	B	33	.012		
10.	Hydrogen Drying (Molecular Sieves)	B	32	.0117		
11.	Crude TCS Recycle System	B	58	.0212		
12.	Silicon Fines Separation	B	15	.0055		
13.	Material Handling	A			90	.0329
14.	Polysilicon Production		720	.2628		
	TOTAL		1303	.4758	90	.0329

NOTES:

1. A Batch Process or Multiple Small Units
   B Average Process
   C Automated Process

2. Man hours/day Unit from Figure 4-6, Peters and Timmerhaus (7).

3. Polysilicon manpower requirements based on batch operation with approximately 1 operator per 10 reactors.

The reaction chemistry (Table IIB-1.2) specifies the majority of components that occur in the reacting unit operations. These were used to obtain the material balance around these stages. This material balance is utilized to calculate the raw material requirements given in Table IIB-1.3. The amount of silicon tetrachloride (TET) by-product calculated is the amount left after complete purification. The major raw material requirements are M.G. silicon and HCl, which are used to generate the intermediate TCS, and hydrogen for the rod reactors. The hydrogen useage is low because most of it is recovered and recycled.

The utility requirements (Table IIB-1.4) are obtained from an energy balance around each piece of major process equipment. Both reaction systems require cooling and the numerous heat exchangers require either steam, cooling water, or refrigerant. The major utility requirement is the electrical requirement for the rod reactors.

The list of major process equipment (62 pieces) is shown in Table IIB-1.5. Columns one and two list the equipment and function as obtained from the detailed process flowsheet. The duty, in column 3, is obtained from Table IIB-1.1 (Base Case Conditions) and the energy balance required to prepare Table (Utility Requirements). Column four (size) was determined by standard chemical engineering design equations for each piece of equipment to match the duty in Column 3. The materials of construction (Column 5) were decided upon after conversations with Dr. Crossman.

The production labor requirements (Table IIB-1.6) were estimated for operations 1 through 13 by the technique described in Peters and Timmerhaus (ref. 7) and utilized for alternate processes already completed. The polysilicon production labor requirements were estimated at one operator per 10 reactors.

These initial results for the conventional polysilicon process are being reviewed primarily in the areas of major process equipment design and production labor requirements. Upon completion of the review, the finalized results will be forwarded for economic analyses to provide estimates of plant investment and production costs for the polysilicon produced by conventional process technology.

III. ECONOMIC ANALYSES (TASK 3)

Economic analyses activities were continued during this reporting period to aid in the cost evaluation of alternate processes under consideration for solar cell grade silicon.

Primary efforts were devoted to the preliminary economic analysis of the conventional polysilicon process. The status, including activities accomplished, in progress, and planned is shown in Table III-1.

The initial results for the preliminary economic analysis are summarized in a tabular format. The guide for the tabular format is given below for the accompanying tables:

1. Process Design Inputs....................Table III-1.1
2. Base Case Conditions.....................Table III-1.2
3. Raw Material Cost........................Table III-1.3
4. Utility Cost.............................Table III-1.4
5. Major Process Equipment Cost.............Table III-1.5
6. Production Labor Cost....................Table III-1.6

The process design inputs are given in Table III-1.1 including raw materials, utilities, equipment and labor requirements. The base case conditions for the preliminary cost analysis are presented in Table III-1.2 including the reference 1975 time period.

The preliminary estimate of cost for raw materials, utilities, major process equipment and labor required for the production of silicon in the conventional polysilicon process are detailed in Table III-1.3 to III-1.6.

In Table III-1.4 for utilities, a value of 3¢/kw-hr for the reference time period was used for electrical cost for industrial power. This value may be slightly high based on a recent plant site survey (ref. 35). The survey indicated an average cost for 1975 of industrial power to be 2.27¢/kw-hr for Arizona, 2.48¢/kw-hr for Michigan and 1.49¢/kw-hr for Texas. A lower electrical power cost (such as 1.5¢/kw-hr versus 3¢/kw-hr) will result in a lower product cost for the polysilicon. This is especially true since the conventional polysilicon process has such high electrical requirements (350-400 kw-hr/KG of silicon) for the production of polysilicon.

Review of these initial results is progress in the areas of major process equipment and production labor requirements and associated costs. Upon completion of the review, major activities in economic analyses will focus on estimates of plant investment and product costs for the production of semiconductor grade polysilicon via the conventional hairpin process technology.

TABLE III-1

ECONOMIC ANALYSES: PRELIMINARY ECONOMIC ANALYSIS ACTIVITIES FOR CONVENTIONAL POLYSILICON PROCESS

Prel. Process Economic Activity	Status
1. Process Design Inputs	●
1. Raw Material Requirements	●
2. Utility Requirements	●
3. Equipment List	●
4. Labor Requirements	●
2. Specify Base Case Conditions	◐
1. Base Year for Costs	◐
2. Appropriate Indices for Costs	◐
3. Additional	◐
3. Raw Material Costs	◐
1. Base Cost/Lb. of Material	◐
2. Material Cost/Kg of Silicon	◐
3. Total Cost/Kg of Silicon	◐
4. Utility Costs	●
1. Base Cost for Each Utility	●
2. Utility Cost/Kg of Silicon	●
3. Total Cost/Kg of Silicon	●
5. Major Process Equipment Costs	◐
1. Individual Equipment Cost	◐
2. Cost Index Adjustment	◐
6. Production Labor Costs	◐
1. Base Cost Per Man Hour	◐
2. Cost/Kg Silicon Per Area	◐
3. Total Cost/Kg Silicon	◐
7. Estimation of Plant Investment	◐
1. Battery Limits Direct Costs	◐
2. Other Direct Costs	◐
3. Indirect Costs	◐
4. Contingency	◐
5. Total Plant Investment (Fixed Capital)	◐
8. Estimation of Total Product Cost	◐
1. Direct Manufacturing Cost	◐
2. Indirect Manufacturing Cost	◐
3. Plant Overhead	◐
4. By-Product Credit	◐
5. General Expenses	◐
6. Total Cost of Product	◐

O Plan
◐ In Progress
● Complete

TABLE III-1.1

PROCESS DESIGN INPUTS FOR
CONVENTIONAL POLYSILICON PROCESS

1. Raw Material Requirements
   -M.G. silicon, anhydrous HCl, caustic, hydrogen, silicon tetrachloride (by-product)
   -see table for "Raw Material Cost"

2. Utility
   -electrical, steam, cooling water, etc.
   -see table for "Utility Cost"

3. Equipment List
   -62 pieces of major process equipment
   -process vessels, heat exchangers, reactor, etc.
   -see table for "Major Process Equipment Cost"

4. Labor Requirements
   -production labor for deposition, vaporization, product handling, etc.
   -see table for "Production Labor Cost"

TABLE III-1.2

BASE CASE CONDITIONS FOR
CONVENTIONAL POLYSILICON PROCESS

1. Capital Equipment
   -January 1975 Cost Index for Capital Equipment Cost
   -January 1975 Cost Index Value = 430

2. Utilities
   -Electrical, Steam, Cooling Water, Nitrogen
   -January 1975 Cost Index (U.S. Dept. Labor)
   -Values determined by literature search and summarized in cost standardization work

3. Raw Material Cost
   -Chemical Marketing Reporter
   -January 1975 Value
   -Other Sources

4. Labor Cost
   -Average for Chemical Petroleum, Coal and Allied Industries (1975)
   -Skilled \$6.90/hr
   -Semiskilled \$4.90/hr

TABLE III-1.3

RAW MATERIAL COST FOR
CONVENTIONAL POLYSILICON PROCESS

Raw Material	Requirement lb/Kg of Silicon	$/lb of Material	Cost $/Kg Of Silicon
1. M.G. Silicon	6.72 (Kg/Kg)	1.0/Kg (Ref.33)	6.72
2. Anhydrous HCl	57.96	.10 (Ref. 34)	5.79
3. Hydrogen	.828	.96 (Ref. 33)	.79
4. Caustic (50% NaOH)	53.29	.0382 (Ref. 12)	2.04
5. $SiCl_4$ (By Product)	46.12	.135 (Ref. 12)	-6.23 (credit)
		TOTAL COST	$ 9.11/Kg Silicon

TABLE III-1.4

UTILITY COST FOR CONVENTIONAL
POLYSILICON PROCESS

	Utility	Requirements/Kg of Silicon	Cost of Utility	Cost $/Kg of Silicon
1.	Electricity	384.6 kw-hr	$ .03/kw-hr	$ 11.54
2.	Steam	152 Pounds	- *	-
3.	Cooling Water	984.5 Gallons	$ .08/M Gal.	.08
4.	Process Water	320.9 Gallons	$ .35/M Gal.	.11
5.	Refrigerant (-40°F)	42.1 M BTU	$10.38/MM BTU	.44
6.	Refrigerant (34°F)	92.3 M BTU	$ 3.75/MM BTU	.35
7.	High Temperature Coolant	582 Pounds	$ 2.7/M Pounds	1.57
8.	Nitrogen	349 SCF	$ .50/M SCF	.17
			TOTAL COST	$14.26/Kg Silicon

NOTES

* All steam produced by cooling jacket on polysilicon rod reactor.

TABLE III-1.5

PURCHASED COST OF MAJOR PROCESS EQUIPMENT FOR CONVENTIONAL POLYSILICON PROCESS

	Equipment	Purchased Cost, $M
1.	(T1) M.G. Silicon Storage Hopper	24.1
2.	(T2) Liquid HCl Storage Tank	435.96
3.	(T3) Crude TCS Hold Tank (3)	178.8
4.	(T4) Waste Hold Tank	14.9
5.	(T5) TCS Reactor Off Gas Flash Tank	7.2
6.	(T6) Hydrogen Storage Tank	152.1
7.	(T7) Polysilicon Storage Space	10.8
8.	(T8) Tet Storage Tanks (2)	85.2
9.	(T9) Tet Feed Tanks (2)	57.8
10.	(T10) TCS Feed Tanks (3)	42.6
11.	(T11) TCS Storage Tanks (3)	127.8
12.	(T12) TET/TCS Feed Tanks (3)	54.
13.	(T13) Caustic Storage Tank	106.7
14.	(T14) #1 Distillation Condenser Flash Tank	.85
15.	(T15) Rod Reactor Off Gas Flash Tank	7.2
16.	(H1) HCl Vaporizer	2.5
17.	(H2) TCS Reactor Off Gas Cooler	7
18.	(H3) TCS Reactor Off Gas Condenser	46.3
19.	(H4) #1 Scrubber Vapor Heater	.75
20.	(H5) #1 Distillation Column Condenser	14.
21.	(H6) #1 Distillation Column Calandria	9.25
22.	(H7) #2 Distillation Column Condenser	14.6
23.	(H8) #2 Distillation Column Calandria	11.92
24.	(H9) #3 Distillation Column Condenser	9.1
25.	(H10) #3 Distillation Column Calandria	5.8
26.	(H11) TCS Vaporizer	1.8
27.	(H12) Rod Reactor Off Gas Cooler	49.4
28.	(H13) Rod Reactor Off Gas Condenser	97.5
29.	(H14) #2 Scrubber Vapor Heater	5.8
30.	(H15) Liquid Recycle Heater	2.3
31.	(H16) #4 Distillation Column Condenser	6.4
32.	(H17) #4 Distillation Column Calandria	3.7
33.	(H18) Nitrogen Heater	1.3

TABLE III-1.5 (Continued)

34.	(P1)	TCS Reactor Off Gas Compressor	53.2
35.	(P2)	Caustic Supply Pump	1.56
36.	(P3)	#1 Distillation Column Overheads Pump	2.64
37.	(P4)	#1 Distillation Column Calandria Pump	3.83
38.	(P5)	TET/TCS Feed Pump	2.04
39.	(P6)	#2 Distillation Column Overhead Pump	2.8
40.	(P7)	TCS Feed Pump	1.8
41.	(P8)	#2 Distillation Column Calandria Pump	3.8
42.	(P9)	#3 Distillation Column Overhead Pump	2.2
43.	(P10)	Rod Reactor TCS Feed Pump	1.7
44.	(P11)	#3 Distillation Column Calandria Pump	2.6
45.	(P12)	Rod Reactor Off Gas Compressor	235.5
46.	(P13)	#4 Distillation Column Overheads Pump	1.87
47.	(P14)	#4 Distillation Column Calandria Pump	1.87
48.	(P15)	TET Feed Pump	1.56
49.	(P16)	Waste Treatment Pump	.77
50.	(P17)	Crude TCS Feed Pump	1.9
51.	(P18)	Process Water Feed Pump	3.7
52.	(C1)	#1 Gas Scrubber	53.2
53.	(C2)	#2 Gas Scrubber	29.
54.	(C3)	#1 Distillation Column	26.1
55.	(C4)	#2 Distillation Column	27.7
56.	(C5)	#3 Distillation Column	8.9
57.	(C6)	#4 Distillation Column	6.7
58.	(R1)	TCS Fluidized Bed Reactor	57.2
59.	(R2)	Polysilicon Rod Reactors (285)	56. (each)
60.	(A1)	Molecular Sieves	16.77
61.	(A2)	Fines Separator	4.8
62.	(A3)	Hydrogen Flare	1.
		TOTAL PURCHASED COST	$18,112.14

TABLE III-1.6

PRODUCTION LABOR COST FOR
CONVENTIONAL POLYSILICON PROCESS

	Unit Operation	Skilled Labor Man-Hrs/Kg Si	Semiskilled Labor Man-Hrs/Kg Si	Cost $/Kg Si
1.	TCS Production	.0292		.2014
2.	Vaporization	.0219		.1511
3.	Vapor Compression	.0219		.1511
4.	Vapor Condensation	.0219		.1511
5.	TCS/TET Separation	.0146		.1007
6.	TCS Purification	.0128		.0883
7.	TET Purification	.011		.0759
8.	Waste Treatment	.0292		.2014
9.	Gas Scrubbing	.012		.0828
10.	Hydrogen Drying (Molecular Sieves)	.0117		.0807
11.	Crude TCS Recycle System	.0212		.1463
12.	Silicon Fines Separation	.0055		.038
13.	Materials Handling		.0329	.1612
14.	Polysilicon Production	.2628		1.8133
			TOTAL COST	$3.44/Kg Silicon

NOTES

Based on labor costs of $6.90 skilled, $4.90 semiskilled.

IV. SUMMARY - CONCLUSIONS

Based on major activities accomplished in this reporting period, the following summary-conclusions are made:

1. Task 1.

Major efforts were continued for process system properties of silicon source materials under consideration for solar cell grade silicon. Primary activities focused on property data for silane.

Liquid viscosity data for silane were correlated as a function of temperature to cover the entire liquid range. Correlation values and data were in good agreement with average absolute deviation of only 1.4%.

Estimates of gas and liquid thermal conductivity results are reported for silane. Unfortunately, there are no experimental data for these transport properties.

For additional silane properties, results are presented for heat and free energy of formation of the gas as a function of temperature. The correlation results are based on data from both American and Russian sources. In general, the agreement of the correlation and data values is quite good. Average deviations are less than 0.30 kcal/g-mol.

2. Task 2

Major efforts were devoted to preliminary process design for the conventional polysilicon process. Engineering design calculations for the preliminary process flowsheet, material balance and energy balance are essentially 100% complete. Major process equipment design and production labor requirements are 80% complete.

Initial results for the conventional polysilicon process indicate the key items are M.G. silicon and HCl consumption; electrical requirements for the rod reactors; and the large number of major equipment items (62). The criteria for design was selected to be commensurate with the design basis for the alternate processes to produce solar cell grade silicon.

Additional chemical engineering activities are being devoted to preliminary process design for the silane (Union Carbide) process. For the process flow diagram as initially received, technical interchange was initiated with Union Carbide to refine the material balance. A revised flowsheet has been received. A preliminary review is essentially complete on several major material balance items. Process design will now proceed.

3. Task 3

Preliminary cost analysis is in progress for the conventional polysilicon process used in the United States and Europe for the production of semiconductor grade polysilicon. Plant investment and product cost will be determined upon completion of review of major process equipment and production labor requirements.

V. PLANS

Plans for the next reporting period are summarized below:

1. Task 1.

Continued analyses of process system properties for silicon source materials under consideration for solar cell grade silicon.

Perform additional correlation activities on experimental data.

2. Task 2.

Design activity on the silane process will proceed utilizing the revised flowsheet received.

Continue preliminary design of the conventional polysilicon process. Complete review of initial results including major process equipment and production labor requirements.

3. Task 3.

Perform preliminary cost analysis for conventional polysilicon process including estimates for plant investment and product costs for semiconductor grade polysilicon.

Perform additional economic analyses on alternate processes under consideration for solar cell grade silicon.

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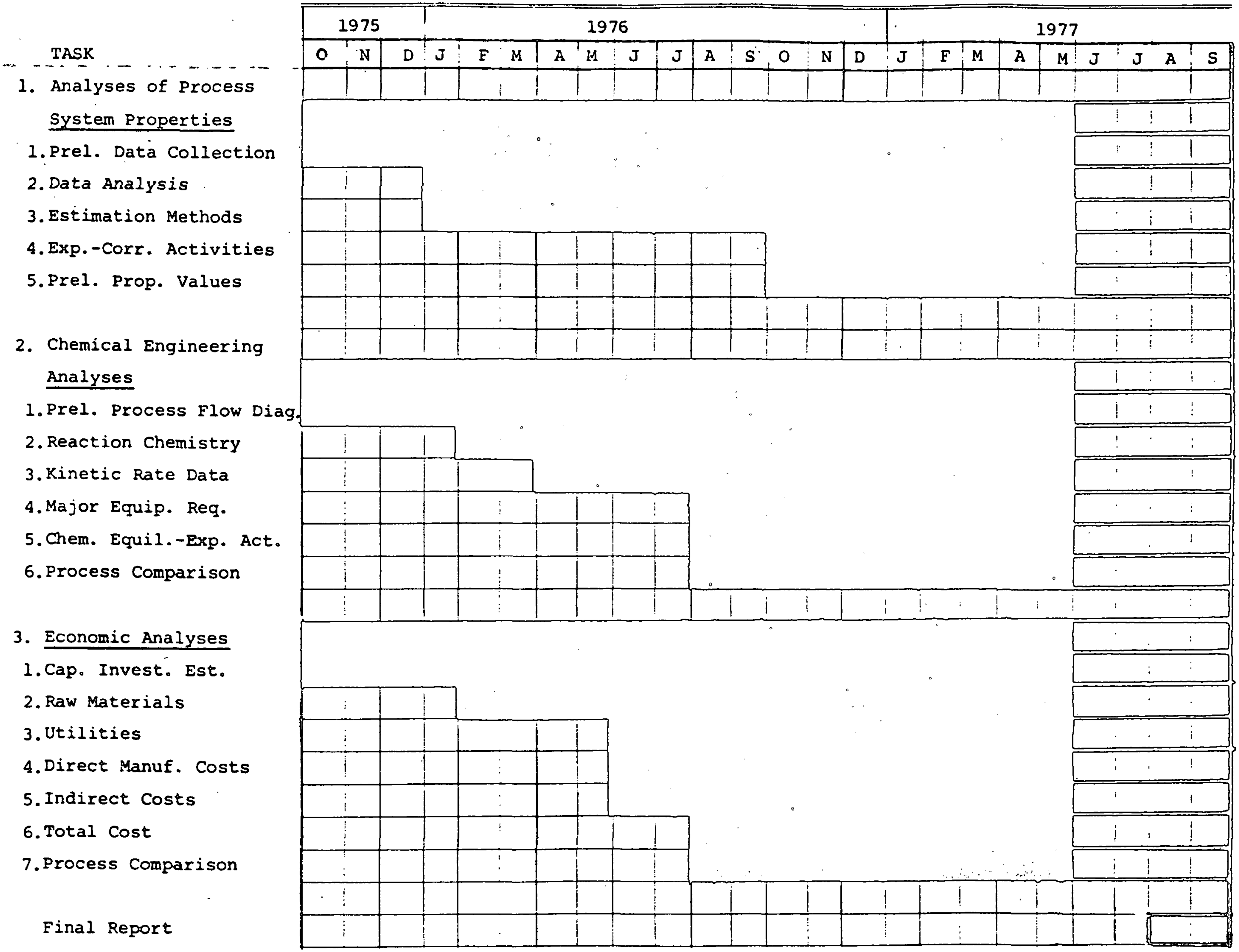
MILESTONE CHART



PROCESS FEASIBILITY STUDY IN
SUPPORT OF SILICON MATERIAL TASK I

JPL Contract No. 954343